

Quarterly Progress Report on
Standard Agreement No. 04-329
For the Period
August 1, 2006 through November 30, 2006

***Development of an Improved VOC Analysis Method for Architectural
Coatings***

Prepared for California Air Resources Board
and the California Environmental Protection Agency

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Disclaimer-

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Acknowledgements

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I. Work This Reporting Period

This report summarizes work performed on the project from August 1, 2006 through November 30, 2006.

A. Task 2 Activities

During this time period, work was continued on Task 2 activities including

- Requesting, cataloging and splitting of samples
- Refinement of testing procedures
- Preliminary analysis of samples

We also continued our communications with other groups involved in VOC analysis and methods. In particular, we participated in phone conferences hosted by the South Coast Air Quality Management District (SCAQMD). These conferences typically included participation by SCAQMD, CARB, EPA and others knowledgeable in the field. These conference calls help us judge the relevance of this project in relation to needs of other constituencies.

B. Samples

Samples of the 86 coatings chosen by CARB for analysis for this project were ordered directly from the manufacturers. To date we have received twenty samples. Of these, one sample, a water-based metallic aluminum pigmented coating, had to be disposed of. After being stored for a few months, the top of the can developed an unusual bulge, and when the top was removed, the coating rapidly expanded. It seems an unwanted reaction had occurred.

A list of the coatings requested is given in Table 1. These samples were typically supplied in one gallon containers. After thorough mixing, each sample was divided into four one quart samples to be used for testing by us and possibly by other laboratories as part of the validation study associated with this project. These samples are in lined one-quart metal paint cans. These samples are currently undergoing analysis. Initially, the density of each coating is determined using a weight per gallon cup. The solids fraction is then determined using ASTM 2369. Great care is taken to insure consistent amounts of coating and water are used in the solids determination since we have determined in previous work that the amount of coating and water can affect the results obtained, especially if high boiling materials are present.

We are in the process of again contacting those manufacturers who have not yet sent samples to determine if they are willing to send samples for analysis

Table 1. Coatings requested from manufacturers for analysis

	Coating Category	WATERBORNE				SOLVENTBORNE			
		Low VOC	High VOC	High Multi	Low Solids	High Multi	Low Solids	High Solids	High Exempt
1	Bituminous Roof	X							
2	Bituminous Roof		X						
3	Bituminous Roof	X							
4	Bituminous Roof	X							
5	Bond Breakers				X				
6	Bond Breakers				X				
7	Bond Breakers				X				
8	Concrete Curing Compounds				X				
9	Concrete Curing Compounds				X				
10	Concrete Curing Compounds				X				
11	Concrete Curing Compounds	X			X				
12	Driveway Sealer	X							
13	Driveway Sealer	X							
14	Driveway Sealer	X							
15	Driveway Sealer	X							
16	Dry Fog	X							
17	Dry Fog	X							
18	Dry Fog	X							
19	Faux Finishing		X						
20	Faux Finishing	X							
21	Faux Finishing		X		X				
22	Fire Resistive	X							
23	Fire Resistive					X			
24	Floor	X							
25	Floor							X	
26	Floor		X						
27	Form Release Compounds							X	
28	Form Release Compounds				X				
29	Form Release Compounds							X	
30	High Temperature								X
31	High Temperature								X
32	Industrial Maintenance					X			
33	Industrial Maintenance					X			
34	Industrial Maintenance					X			
35	Lacquers	X							
36	Lacquers						X		X
37	Lacquers								X
38	Lacquers						X		X
39	Low Solids	X			X				
40	Low Solids	X			X				
41	Magnesite Cement								X
42	Mastic Texture		X						
43	Mastic Texture	X							
44	Mastic Texture	X							
45	Metallic Pigmented	X							
46	Metallic Pigmented	X							
47	Metallic Pigmented		X						
48	Multi-Color	X			X				
49	Multi-Color	X							
50	Multi-Color	X							

Table 1. Coatings requested from manufacturers for analysis (con't.)

	Coating Category	WATERBORNE				SOLVENTBORNE			
		Low VOC	High VOC	High Multi	Low Solids	High Multi	Low Solids	High Solids	High Exempt
51	Quick Dry Primer, Sealer, and Undercoater	X							
52	Recycled	X							
53	Roof	X							
54	Roof	X							
55	Roof	X							
56	Rust Preventative		X						
57	Sanding Sealers				X				
58	Shellacs - Clear						X		
59	Shellacs - Clear						X		
60	Specialty Primer, Sealer, and Undercoater	X							
61	Stains - Clear/Semitransparent				X				
62	Stains - Clear/Semitransparent				X				
63	Stains - Clear/Semitransparent								X
64	Stains - Opaque	X							
65	Stains - Opaque	X			X				
66	Swimming Pool					X			
67	Swimming Pool			X					
68	Swimming Pool					X			
69	Traffic Marking								X
70	Traffic Marking								X
71	Traffic Marking	X							
72	Varnishes - Clear		X	X					
73	Varnishes - Clear		X						
74	Varnishes - Clear			X					
75	Varnishes - Clear		X						
76	Varnishes - Semitransparent		X						
77	Varnishes - Semitransparent				X				
78	Waterproofing Concrete/Masonry Sealers	X							
79	Waterproofing Concrete/Masonry Sealers	X							
80	Waterproofing Concrete/Masonry Sealers	X			X				
81	Waterproofing Sealers	X							
82	Waterproofing Sealers				X				
83	Waterproofing Sealers	X			X				
84	Wood Preservatives	X			X				
85	Wood Preservatives				X				
86	Wood Preservatives	X			X				

C. Refinement of Testing Procedures

We have begun direct VOC analysis of waterborne coatings using a modification of ASTM 6886. To date we have analyzed five coatings, listed below in Table 2, and the results for these coatings are given later in this report. The coating ID is for internal use only and does not correspond to the number listed in Table 1.

Table 2. Coatings Analyzed

Cal Poly Coating ID	Coating Type
34	Dry Fog
35	Dry Fog
38	Waterproofing Concrete/Masonry Sealers
43	Roof
50	Mastic Texture

Sample Preparation for Direct GC/FID or GC/MS and Static Headspace Analysis

To prepare samples, 1.5 to 2.0g of the neat liquid coating, weighed to 0.1mg, is placed in a 40mL vial containing 2 to 3g of ceramic beads. 10.0 mL of an aqueous solution of 0.1% ethylene glycol diethyl ether (EGDE) is added and the contents are mixed by shaking to obtain homogeneity. EGDE serves as the internal standard. EGDE is water soluble, is not used in coatings formulation, and has a retention time distinct from any known VOCs. We have found water to be an acceptable solvent for direct analysis of waterborne coatings. This avoids the use of tetrahydrofuran (THF). The ceramic beads function as a mixing aid. After mixing, a small sample (typically 20 μ L) is transferred to a 20mL headspace vial and closed with a crimp cap. This sample is used for obtaining static headspace data after a specified equilibration time at a specified temperature. To prepare a sample for direct GC/FID or GC/MS analysis, the well-mixed aqueous coating solution/dispersion is diluted with an equal volume of acetone in a separate small vial. If the sample contained acetone, an alternate dilution solvent could be used.

Direct GC Analysis

Temperatures: Inlet 260°C, initial temperature 50°C, initial time 4 min, rate 20°C/min, final temperature 260°C, final time 10 min, FID detector 260°C

Split: GC/MS 100 to 1; GC/FID 50 to 1

Flow: GC/MS 0.8 mL/min, GC/FID 1.0 mL/min

Columns:

GC/MS, DB-5, 30m x 0.25mm, 0.25 μ m film

GC/FID, DB-5, 30m x 0.25mm, 1.0 μ m film

Injection volume: 1 μ L

Static Headspace Analysis

Two 20 μ L aliquots of the aqueous solution described above are placed in separate 20mL crimp vials and sealed with crimp caps. One of the samples is equilibrated at 110⁰C for 30 minutes and is then analyzed by GC/FID using the conditions described above. The second sample is equilibrated at 150⁰C for 15 minutes and is then analyzed by GC/FID using the same conditions.

Response factors

To prepare standards, samples of pure compounds likely to be found in the coatings were used. Mixtures prepared from known masses of each compound including internal standard (EGDE) were used to obtain responses factors for each compound for both FID and MS detection. Replicate runs were performed and the results averaged to obtain response factors used in the analyses described in this report. Typical response factors for MS detection are shown below in Table 3.

Table 3. MS Response Factors

ret time/min	compound	MS RF
2.608	1-butanol	0.78
2.893	ethylene glycol	0.28
3.533	propylene glycol	0.46
4.789	EGDE	1.00
5.543	diacetone alcohol	0.87
6.028	p-xylene	2.08
6.56	butoxyethanol	0.99
7.829	Dipropylene glycol monomethyl ether (DPM)	1.08
8.104	2-ethylhexanol	1.45
9.609	butoxyethoxyethanol	0.89
11.052	TEXANOL	1.56
12.758	benzophenone	2.32
13.135	Velate 368	2.20
15.391	dioctyl maleate	2.12
15.617	tributyl phosphate	0.83

D. Results

Each of the five coatings samples was analyzed using both headspace/FID and direct injection GC/MS. Headspace runs were performed using both 110⁰C/30 minute and 150⁰C/15 minute equilibrations. Appropriate response factors were used to determine the fraction of each volatile component in the coating. Each coating contained very small amounts of unidentified VOCs. These were assigned response factors of one. Some additional compounds were identified by

mass spectrometry for which we have not determined response factors. A response factor for a similar known compound was used for these substances. In all cases the possible errors introduced by not knowing the experimental value for the response factors of these compounds are small since the compounds were present in very small amounts, generally contributing much less than 1 g/L to the coating VOC. The results of the analyses are presented in Tables 4-8.

Table 4. Results for Sample 34 Dry Fog Coating

34		Dry Fog					material VOC, g/L	coating VOC, g/L	Cumulative Material VOC	Cumulative Coating VOC	Headspace, fraction, 110C for 30 min (% recovery)	Headspace, fraction, 150C for 15 min (% recovery)
peak	R.T. min	area % of total	Compound	mg	fraction in paint sample	% of total volatile fraction						
1	3.622	1.61%	propylene glycol	5.20	0.0027	5.60	3.36	7.72	3.36	7.72		
2	4.792	6.73%	EGDE (Internal Standard)	10.05	0.0000	0.00	0.00	0.00	3.36	7.72		
3	5.173	0.08%	UK	0.13	0.0001	0.14	0.08	0.19	3.44	7.91		
4	5.569	11.39%	diacetone alcohol	19.66	0.0102	21.19	12.72	29.20	16.16	37.11		
5	5.983	0.41%	UK	0.61	0.0003	0.65	0.39	0.90	16.56	38.01		
6	7.95	2.19%	DPM	3.03	0.0016	3.26	1.96	4.50	18.51	42.51		
7	8.098	1.95%	2-ethylhexanol	2.01	0.0010	2.17	1.30	2.99	19.82	45.49		
8	9.606	1.13%	butoxyethoxy ethanol	1.88	0.0010	2.03	1.22	2.79	21.03	48.29		
9	10	0.75%	hexamethylene tetramine	1.12	0.0006	1.21	0.73	1.67	21.76	50		
10	11.052	28.14%	TEXANOL	26.93	0.0140	29.02	17.42	39.99	39.18	90	84	93
11	13.853	0.06%	UK	0.09	0.0000	0.10	0.06	0.14	39.23	90.08		
12	15.384	2.27%	dioctyl fumarate	1.60	0.0008	1.72	1.03	2.37	40.27	92.45	0	0
13	15.807	41.32%	dioctyl maleate	29.13	0.0151	31.40	18.85	43.27	59.11	135.72	0	0
14	17.05	1.98%	dioctyl adipate	1.40	0.0007	1.50	0.90	2.07	60.02	138	0	0
Total					0.0481	100.00	60	138				

Density lbs/gal g/L	10.4103	1247
Solids fraction		0.5160
Paint weight, mg		1928
Total paint VOC wt fraction		0.0314
Calculated water wt fraction		0.4526
Material VOC, g/L		39
Coating VOC, g/L		90
Lbs solids/gal		5.37
REPORTED VALUE, Coating VOC (can)		72

Table 5. Results for Sample 35 Dry Fog Coating

35	Dry Fog											
peak	R.T. min	area % of total	Compound	mg	fraction in paint sample	% of total volatile fraction	material VOC, g/L	coating VOC, g/L	Cumulative Material VOC	Cumulative Coating VOC	Headspace, fraction, 110C for 30 min (% recovery)	Headspace, fraction, 150C for 15 min (% recovery)
1	3.002	0.26%	UK	0.15	0.0001	0.31	0.11	0.26	0.11	0.26		
2	3.52	0.28%	UK	0.17	0.0001	0.33	0.12	0.28	0.23	0.54		
3	3.635	9.92%	propylene glycol	12.64	0.0066	25.39	8.92	21.18	9.14	21.71		
4	3.805	0.17%	UK	0.10	0.0001	0.20	0.07	0.16	9.21	21.88		
5	4.792	17.08%	EGDE (Internal Standard)	10.05	0.0000	0.00	0.00	0.00	9.21	21.88		
6	5.189	0.13%	UK	0.08	0.0000	0.16	0.06	0.13	9.27	22.01		
7	5.553	18.22%	diacetone alcohol	12.39	0.0065	24.90	8.75	20.77	18.01	42.78		
8	6.002	1.05%	UK	0.62	0.0003	1.24	0.43	1.03	18.45	43.81		
11	7.96	0.65%	dipropyleneglycol methyl ether	0.38	0.0002	0.77	0.27	0.64	18.72	44.45		
12	8.101	1.82%	2-ethylhexanol	0.74	0.0004	1.49	0.52	1.24	19.24	45.69		
13	9.259	0.33%	UK	0.19	0.0001	0.38	0.14	0.32	19.38	46.01		
14	9.357	0.74%	UK	0.44	0.0002	0.88	0.31	0.73	19.68	46.75		
15	9.609	6.90%	butoxyethoxy ethanol	4.54	0.0024	9.13	3.21	7.61	22.89	54.36		
16	9.705	1.35%	UK	0.79	0.0004	1.59	0.56	1.33	23.45	55.69		
17	9.777	0.43%	UK	0.25	0.0001	0.51	0.18	0.43	23.63	56.11		
18	9.829	0.72%	UK	0.42	0.0002	0.85	0.30	0.71	23.93	56.83		
19	9.918	0.74%	UK	0.43	0.0002	0.87	0.31	0.73	24.23	57.55		
20	10.003	3.01%	hexamethylene tetramine	1.77	0.0009	3.56	1.25	2.97	25.48	61		
21	11.052	36.22%	TEXANOL	13.66	0.0072	27.44	9.64	22.89	35	83	71	72
			Total		0.0262	100.00	35	83				

Density lbs/gal g/L	11.21	1343
Solids fraction		0.5426
Paint weight, mg		1902.6
Total paint VOC wt fraction		0.0262
Calculated water wt fraction		0.4312
Material VOC, g/L		35
Coating VOC, g/L		83
Lbs solids/gal		6.08
REPORTED VALUE, Coating VOC (can)		66

Table 6. Results for Sample 38 Waterproofing Concrete/Masonry Sealer

38 Waterproofing Concrete/Masonry Sealer												
peak	R.T. min	area % of total	Compound	mg	fraction in paint sample	% of total volatile fraction	material VOC, g/L	coating VOC, g/L	Cumulative Material VOC	Cumulative Coating VOC	Headspace, fraction, 110C for 30 min (% recovery)	Headspace, fraction, 150C for 15 min (% recovery)
1	2.611	3.20%	1-butanol	1.80	0.0008	2.72	1.05	2.16	1.05	2.16		
2	3.054	34.19%	ethylene glycol	53.63	0.0238	81.02	31.13	64.36	32.18	66.52		
3	3.664	1.02%	UK	0.45	0.0002	0.68	0.26	0.54	32.44	67.06		
4	4.628	1.69%	UK	0.74	0.0003	1.12	0.43	0.89	32.87	67.95		
5	4.795	22.95%	EGDE (Internal Standard)	10.05		0.00	0.00	0.00	32.87	67.95		
6	5.605	2.09%	diacetone alcohol	1.06	0.0005	1.60	0.61	1.27	33.48	69.22		
7	6.028	8.36%	p-xylene	1.76	0.0008	2.66	1.02	2.12	34.51	71.34		
8	6.228	1.32%	n-butyl ether	0.58	0.0003	0.87	0.34	0.69	34.84	72.03		
9	6.369	0.96%	styrene	0.42	0.0002	0.63	0.24	0.50	35.09	72.54		
10	6.612	0.51%	butyl propionate	0.22	0.0001	0.34	0.13	0.27	35.22	72.80		
11	9.609	1.32%	butoxyethoxy ethanol	0.65	0.0003	0.98	0.38	0.78	35.59	73.58		
12	9.996	2.03%	hexamethylene tetramine	0.89	0.0004	1.34	0.52	1.06	36.11	75		
	11.052		(TEXANOL)	0.00	0.0000	0.00	0	0	36	75		
13	12.754	6.55%	benzophenone	1.24	0.0005	1.87	0.72	1.48	36.82	76.13	77	117
14	13.036	0.67%	UK	0.29	0.0001	0.45	0.17	0.35	37.00	76.48		
15	13.849	13.14%	tetrachloro phthalonitrile	2.47	0.0011	3.72	1.43	2.96	38.43	79	11	39
Total					0.0294	100.00	38	79				

Density lbs/gal g/L	10.93	1309
Solids fraction		0.5780
Paint weight, mg		2254.9
Total paint VOC wt fraction		0.0276
Calculated water wt fraction		0.3944
Material VOC, g/L		36
Coating VOC, g/L		75
Lbs solids/gal		6.32
REPORTED VALUE, Coating VOC		Not reported

Table 7. Results for Sample 43 Roof Coating

43 Roof												
peak	R.T. min	area % of total	Compound	mg	fraction in paint sample	% of total volatile fraction	material VOC, g/L	coating VOC, g/L	Cumulative Material VOC	Cumulative Coating VOC	Headspace, fraction, 110C for 30 min (% recovery)	Headspace, fraction, 150C for 15 min (% recovery)
1	2.739	1.35%	UK	0.52	0.0003	1.63	0.34	0.91	0.34	0.91		
2	3.018	9.57%	ethylene glycol	13.25	0.0069	41.49	8.54	23.06	8.88	23.97		
3	4.795	26.01%	EGDE (Internal Standard)	10.05		0.00	0.00	0.00	8.88	23.97		
4	5.592	13.44%	diacetone alcohol	6.00	0.0031	18.80	3.87	10.45	12.75	34.42		
5	6.225	0.62%	UK	0.24	0.0001	0.74	0.15	0.41	12.90	34.84		
6	6.612	0.17%	UK	0.06	0.0000	0.20	0.04	0.11	12.94	34.95		
7	10	0.96%	hexamethylene tetramine	0.37	0.0002	1.16	0.24	0.64	13.18	36		
8	11.052	40.97%	TEXANOL	10.14	0.0053	31.76	6.54	17.65	20	53	95	108
9	12.754	6.03%	benzophenone	1.00	0.0005	3.14	0.65	1.75	20.37	54.99	73	124
10	15.804	0.89%	UK	0.34	0.0002	1.08	0.22	0.60	20.59	56		
Total					0.0166	100.00	21	56				

Density lbs/gal g/L	10.33	1237
Solids fraction		0.4752
Paint weight, mg		1918.9
Total paint VOC wt fraction		0.0159
Calculated water wt fraction		0.5088
Material VOC, g/L		20
Coating VOC, g/L		53
Lbs solids/gal		4.91
REPORTED VALUE, Coating VOC (tech. data sheet)		<50

Table 8. Results for Sample 50 Mastic Texture Coating

50 Mastic Texture												
peak	R.T. min	area % of total	Compound	mg	fraction in paint sample	% of total volatile fraction	material VOC, g/L	coating VOC, g/L	Cumulative Material VOC	Cumulative Coating VOC	Headspace, fraction, 110C for 30 min (% recovery)	Headspace, fraction, 150C for 15 min (% recovery)
1	2.611	1.08%	1-butanol	0.61	0.0003	1.03	0.45	0.83	0.45	0.83		
2	3.667	53.92%	propylene glycol	50.72	0.0271	86.45	37.83	69.61	38.29	70.44		
3	4.792	23.13%	EGDE (Internal Standard)	10.05	0.0000	0.00	0.00	0.00	38.29	70.44		
4	5.56	7.83%	diacetone alcohol	3.93	0.0021	6.70	2.93	5.39	41.22	75.83		
5	6.229	1.16%	n-butyl ether	0.50	0.0003	0.86	0.38	0.69	41.59	77		
	11.052		(TEXANOL)			0.00	0	0	42	77		
6	12.754	6.09%	benzophenone	1.14	0.0006	1.94	0.85	1.56	42.44	78.09	36	69
7	12.971	0.55%	UK	0.24	0.0001	0.40	0.18	0.33	42.62	78.41		
8	13.036	1.26%	UK	0.55	0.0003	0.93	0.41	0.75	43.03	79.16		
9	13.479	4.77%	(4-methylphenyl) phenylmethanone	0.89	0.0005	1.52	0.67	1.23	43.69	80.39	28	66
10	14.145	0.21%	UK	0.09	0.0000	0.16	0.07	0.13	43.76	81		
Total					0.0314	100.00	44	81				

Density lbs/gal g/L	11.63	1394
Solids fraction		0.6427
Paint weight, mg		1868.3
Total paint VOC wt fraction		0.0298
Calculated water wt fraction		0.3275
Material VOC, g/L		42
Coating VOC, g/L		77
Lbs solids/gal		7.48
REPORTED VALUE, Coating VOC		60

For each coating we have listed the contribution to material and coating VOC for each compound and the cumulative material and coating VOC based on retention time. In this way the effect on total VOC of excluding compounds with retention times longer than some specified time (for example, that of Texanol) can be determined. We have also listed the fraction of known higher boiling compounds (those with retention times equal to or longer than that for Texanol) detected using headspace analysis. We determined the percentage retained under two different equilibration conditions: 110°C for 30 minutes and 150°C for 15 minutes. Equilibration at 110° should approximate conditions used for determining the solids fraction using ASTM 2369. Under the heading *Cumulative Coatings VOC* we have highlighted the VOC value up to and including Texanol and the total value including all compounds (these highlighted values have been rounded to the nearest gram per liter). We have also reported the Material and Coating VOC in g/L in the summary table at the bottom of the larger table including all VOCs with retention times up to and including that of Texanol. We have chosen the Texanol retention time as the cutoff since Texanol is often considered a marker compound between those compounds that do and do not contribute to VOC when measured using EPA Method 24.

E. Discussion

These coatings represent only a small fraction of those to be studied in this project, however they show several significant differences from the low VOC waterborne coatings for which ASTM Method 6886 was developed. ASTM 6886 was developed primarily for interior and exterior architectural coatings with a small number of possible VOCs. For this study we have modified ASTM 6886 by using water as the solvent and ethylene glycol diethyl ether (EGDE) as the internal standard. We have also slightly modified the method of sample preparation so both headspace and direct injection samples can be prepared.

We have identified several VOCs in these coatings not typically found in interior and exterior architectural coatings. These include alcohols, amines, glycol ethers, aromatics and high boiling esters. Compounds detected having retention times longer than that for Texanol include dioctyl maleate, dioctyl fumarate, dicotyl adipate, benzophenone, tetrachlorophthalonitrile and (4-methylphenyl)phenylmethanone. The only one of these higher boiling compounds present in a significant amount was dioctyl maleate. We have seen this compound in several other coatings where it is sometimes used instead of or in addition to Texanol.

By analyzing these coatings using static headspace with FID detection we can study the effect of equilibration temperature on amount of higher boiling VOC compound detected. For Texanol, amounts ranging from 71% to 100% of the total Texanol were detected, even at 110°C. This suggests most of the Texanol would be detected for these coatings using EPA Method 24 analysis. For the higher boiling dioctyl esters, none of the compounds were detected using headspace analysis at either equilibration temperature. This suggests these compounds would not be VOCs when analyzed using EPA Method 24. Benzophenone was present in two of the coatings. For both coatings, approximately 70% was detected when equilibrated at 110°C

compared to 100% when equilibrated at 150°C. This suggests most of the benzophenone would be counted as a VOC using EPA Method 24 analysis.

Our analysis of these coatings confirms the validity of using a modified ASTM 6886 method. The incorporation of static headspace analysis allows a determination of those compounds likely to be measured using EPA Method 24. None of these coatings contain reactive materials and all are one component coatings. During the next reporting period we will include tests on two component coatings to assess the applicability of these methods.

II. Future work

We will continue work on Task 2 during the next reporting period. We will continue our efforts to secure the remaining samples and will expand our analyses to other types of coatings included in the sample inventory.

III. Overall progress of project.

Project is on time and on budget.